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पैट्रोलियम और उसके उत्पादों की परीक्षण पद्धतियां

[पी : 146]

निम्न तापमान पर इंजन तेल का लब्ध प्रतिबल और दृश्य श्यानता का निर्धारण

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 146]

DETERMINATION OF YIELD STRESS AND APPARENT VISCOSITY OF ENGINE OILS AT LOW TEMPERATURE

ICS 75.080

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1998 **Price Group 4**

Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This test method is essentially based on ASTM D 4684-92 'Standard test method for determination of yield stress and apparent viscosity of engine oil at low temperature', issued by the American Society for Testing and Materials (USA). Use also be made of the method given in ASTM D 3829-87 as a screening method to predict the pumpability characteristics of engine oils when developing a new oil, since no single method is capable of detecting all potential pumpability problems.

In reporting the results of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).'

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 146]

DETERMINATION OF YIELD STRESS AND APPARENT VISCOSITY OF ENGINE OILS AT LOW TEMPERATURE

1 SCOPE

1.1 This test method covers the measurement of the yield stress and viscosity of engine oils after cooling at controlled rates over a period exceeding 45 h to a final test temperature between –15 and –35°C. The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to 15 s^{-1}.

1.2 The applicability to petroleum products other than engine oils has not been determined.

1.3 This test method uses the millipascal second (mPa.s) as the unit of viscosity. For information, the equivalent SI unit, the millipascal, is shown in parentheses.

1.4 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 TERMINOLOGY

2.1 For the purpose of this method the following definitions shall be apply.

2.1.1 *Apparent Viscosity*

The determined viscosity obtained by use of this method.

2.1.2 *Newtonian Oil or Fluid*

An oil or fluid that at a given temperature exhibits a constant viscosity at all shear rates or shear stresses.

2.1.3 *Non-Newtonian Oil or Fluid*

An oil or fluid that at a given temperature exhibits a viscosity that varies with changing shear stress or shear rate.

2.1.4 *Shear Rate*

The velocity gradient in fluid flow. For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner cylinder surface (such as this apparatus, described in **5.1**), and ignoring any end effects, the shear rate is given as follows:

$$G_r = \frac{2\,(\Omega)\,R_s^2}{(R_s^2 - R_r^2)} = \frac{4\,(\pi)\,R_s^2}{t\,(R_s^2 - R_r^2)}$$

where

G_r = shear rate at the surface of the rotor in reciprocal seconds, s^{-1};

Ω = angular velocity, rad/s;

R_s = stator radius, mm;

R_r = rotor radius, mm; and

t = time in seconds for one revolution of the rotor.

For the specific apparatus being described in **5.1.1**.

$$G_r = 63/t$$

2.1.5 *Shear Stress*

The motivating force per unit area for fluid flow. For the rotary viscometer being described, the rotor surface is the area under shear or the shear area.

$$T_r = 9.81\,M\,(R_o + R_t) \times 10^{-6}$$

$$S_r = \frac{T_r}{2\,\pi\,R_r^2 h} \times 10^9$$

where

T_r = torque applied to rotor, Nm;

M = applied mass, g;

R_o = radius of the shaft, mm;

R_t = radius of the string, mm;

S_r = shear stress at the rotor surface, Pa; and

h = height of the rotor, mm.

For the dimensions given in **5.1.1**.

$$T_r = 31.7\,M \times 10^{-6}$$

$$S_r = 3.5\,M$$

2.1.6 *Viscosity*

The ratio between the applied shear stress and rate of shear, sometimes called the coefficient of dynamic viscosity. This value is thus a measure of the resistance to flow of the liquid. The SI unit of viscosity is the pascal second (Pa.s). A centipoise (cP) is one millipascal second (mPa.s).

2.2 Descriptions of Terms Specific to This Standard

2.2.1 *Borderline Pumping Temperature*

The maximum temperature at which the critical yield stress (35 Pa) or the critical viscosity (30 000 cP) occurs, whichever is the higher temperature.

2.2.2 *Calibration Oil*

Those oils that establish the instrument's reference framework of apparent viscosity *versus* speed, from which the apparent viscosities of test oils are determined. Calibration oils, which are essentially Newtonian fluids, are available commercially, and have an approximate viscosity of 30 000 cP (30 Pa.s) at – 20°C[1].

2.2.3 *Test Oil*

Any oil for which the apparent viscosity and yield stress are to be determined by this test method.

2.2.4 *Yield Stress*

The shear stress required to initiate flow. For oil Newtonian fluids and some non-Newtonian fluids the yield stress is zero. An engine oil can have a yield stress that is a function of its low-temperature cooling rate, soak time, and temperature. Yield stress measurement by this method determines only whether the test oil has a yield stress of at least 35 Pa; a yield stress below 35 Pa is considered to be insignificant.

3 OUTLINES OF TEST METHOD

An engine oil sample is held at 80°C and then cooled at a programmed cooling rate to a final test temperature. A low torque is applied to the rotor shaft to measure the yield stress. A higher torque is then applied to determine the apparent viscosity of the sample.

4 SIGNIFICANCE AND USE

4.1 When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this laboratory test an engine oil is slowly cooled through a temperature range where wax crystallization is known to occur, followed by relatively rapid cooling to the final test temperature. These laboratory test results have predicted as failures the known engine oils that have failed in the field due to the lack of oil pumpability[1]. These documented field failing oils have also consisted of oils normally tested at –25°C. These field failures are believed to be the result of the oil forming a gel structure that results in excessive yield stress of viscosity of the engine oil, or both.

4.2 Cooling Profiles

For oils to be tested at –20°C or colder, Table 1 applies. The cooling profile described in Table 1 is based on the viscosity properties of the ASTM Pumpability Reference Oils (PRO). This series of oils includes oils with normal low temperature flow properties and oils that have been associated with low temperature pumpability problems. (1) through (5). No significance has been determined for the –35°C temperature profile because of the absence of appropriate reference oils.

For oils to be tested at –15° or –10°C, Table 2 applies. Currently, no significance has been determined for this temperature profile because of absence of appropriate reference oils. Similarly, precision of the test method using this profile for the –10°C test temperature is unknown. The temperature profile of Table 2 is derived from the one in Table 1 and has been moved up in temperature relative to Table 1, in consideration of the expected higher cloud points of the viscous oils tested at –15°C and –10°C.

5 APPARATUS

5.1 Mini-Rotary Viscometer

An apparatus that consists of one or more viscometric cells in a temperature-controlled aluminium block. Each cell contains a calibrated rotor-stator set. Rotation of the rotor is achieved by an applied load acting through a string wound around the rotor shaft.

5.1.1 The mini-rotary viscometric cell has the following typical dimensions in millimetres·

Diameter of rotor	17.0
Length of rotor	20.0
Inside diameter of cell	19.0
Radius of shaft	3.18
Radius of string	0.05

5.2 Temperature control system that will operate the heater in the mini-rotary viscometer block and regulate the coolant flow to the block. This controller must regulate the temperature to within the temperature limits described in Table 1 or Table 2.

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[1] Available from Cannon Instrument Co, P.O. Box 16, State College, PA 16804, USA.

[1] Pumpability Reference Oils (PRO) 21 through 29.

5.2.1 Temperature controller is the most critical part of this procedure. The temperature control system can be a single-loop programmable controller to control the block temperature. A process controller that has a proportional band with integral reset and derivative rate control, sometime referred to as a PID controller, is suitable for controlling the temperature. This programmable controller has one control loop and one temperature sensor that provides the appropriate information to the controller to hold the temperature at the programmed set temperature. It has an internal clock that controls the execution of the programme. The controller must be connected so that only heat is supplied to the block during the first 2 h and 20 min of the temperature profile described in Tables 1 and 2. The temperature control during the remaining portion of the temperature profile must be obtained by controlling the coolant flow. This control system must have a minimum temperature sensitivity of 0.1°C and be able to change the temperature at a prescribed rate. When the control system's proportional band, integral (reset), and derivative (rate) parameters are optimized the temperature excursions above and below the profile must be no greater than 0.2°C at a temperature below –5°C. The temperature sensor can be a platinum resistance thermal detector, a thermistor, or a thermocouple. A platinum resistance thermal detector or thermistor sensor is preferred. A 3.2 mm diameter temperature probe can be installed directly into the 3.2 mm diameter well located at the back of the block between cells Nos. 4 and 6. Alternatively, the temperature sensor can be inserted into one of the thermometer wells.

NOTES

1 The sensor is placed in the same unit that is being controlled. The sensor should be placed in the block if the supply of coolant is being controlled. Alternatively, the sensor would be placed in the bath if the bath temperature was being controlled. Do not try to control the block temperature by sensing the block temperature and controlling the refrigeration system.

2 An internally delayed start for the controller is a desirable feature, since this will allow starting the temperature profile unattended.

5.2.2 *Temperature Profile*

The temperature profile is fully described in Tables 1 and 2.

5.3 Thermometers for measuring the temperature of the block. Two are required, one graduated from at least +70 to 90°C in 1°C subdivisions, the other with a scale from at least –36 to +5°C in 0.2°C subdivisions. Other thermometric devices of equal accuracy and resolution may be used to calibrate the temperature sensor.

5.4 Refrigeration Device, consisting of a means of maintaining a coolant (such as methanol) to at least 10°C below the lowest test temperature.

5.5 Circulating System[1] that will circulate the liquid coolant to the block as needed. Methanol is a suitable coolant. One should observe toxicity and flammability precautions that apply to the use of methanol. The circulating system must be capable of maintaining test temperature during the test. If methanol is leaking from the system, discontinue the test and repair the leak.

WARNING — Methanol is flammable.

5.6 Chart Recorder

To verify that the correct cooling curve is being followed, it is recommended that a chart recorder be used to monitor the block temperature.

6 REAGENTS AND MATERIALS

6.1 Newtonian Oil[1]

A low cloud-point of approximately 30 000 cP (30 Pa.s) viscosity at –20°C for calibration of the viscometric cells.

6.2 Methanol

Commercial or technical grade of dry methanol is suitable for the cooling bath.

6.3 Oil Solvent

Commercial heptane or similar solvent that evaporates without leaving a residue is suitable.

WARNING — Flammable.

6.4 Acetone

A technical grade of acetone is suitable provided it does not leave a residue upon evaporation.

WARNING — Flammable.

7 SAMPLING

A representative sample of test oil free from suspended solid material and water is necessary to obtain valid viscosity measurements. If the sample in its container is received below the dew-point temperature of the room, allow the sample to warm to room temperature before opening the container.

8 CALIBRATION AND STANDARDIZATION

8.1 Calibrate the temperature sensor while attached to the temperature controller. The sensed temperature should be verified using the +5 to –36°C reference thermometer noted in **5.3** at a minimum of three temperatures. Make these temperature measurements at least 5°C apart to establish a calibration curve for this combination of temperature sensor and controller. Recheck the temperature sensor periodically using the ice point of water as the reference point. If the corrected sensor temperature deviates from the ice point by more than 0.1°C, repeat the calibration in accordance with this paragraph.

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[1] Available from Cannon Instrument Co, P.O. Box 16, State College, PA 16804, USA.

NOTE — All temperatures in this test method refer to the actual temperature and not necessarily the indicated temperature.

8.2 The calibration of each viscometric cell (viscometer constants) can be determined with the viscosity standard and the following procedure at –20 ± 0.2°C.

8.2.1 Use steps given in **9.2** through **9.2.5**.

8.2.2 Programme the temperature controller to cool the mini-rotary viscometer block to –20°C within 1 h or less, then start the programme.

8.2.3 Allow the oil in the cells to soak at –20 ± 0.2°C for at least 1 h making small temperature adjustment, if necessary, to maintain the test temperature.

8.2.4 At the end of the soak period record the temperature reading (test temperature) and remove the cover of the viscometer cell.

8.2.5 Proceed to steps given in **9.3.1** through **9.3.3**.

8.2.6 Perform step given in **9.4.1**.

8.2.7 Repeat **8.2.5** and **8.2.6** for each of the remaining cells, taking the cells in order from left to right.

8.2.8 Calculate the viscometer constant for each cell (rotor/stator combination) with the following equation:

$$C = n_o/t$$

where

n_o = viscosity of the standard oil, cP (mPa.s) at –20°C,

C = cell constant with 150 g mass Pa, and

t = time in seconds for three complete revolutions.

8.3 It is essential that the ice point of the calibrated thermometer and the temperature sensor are initially measured and periodically measured thereafter. If any change in calibration is noted in the ice point the temperature controller/sensor combination, recalibrate in accordance with **8.1**.

8.4 Check the cooling rate periodically to ensure that the programmed cooling follows the profile described in Table 1 or Table 2. This should be done at least monthly by recording the block temperature as measured by the temperature controller and a thermometer placed in the block at least 3 times during normal working hours. Continual monitoring of the temperature profile is strongly recommended during a cooling programme below 0°C, since deviations from the prescribed temperature profile. Table 1 or Table 2 can cause erroneous test results. This can be achieved through the use of a strip chart recorder or a data acquisition system that periodically (every 1 to 2 min) records the time and block temperature. The allowable temperature deviations are shown in Table 1 or Table 2 and are also outlined in **5.2.**

9 PROCEDURE

9.1 Programme the temperature controller to control the mini-rotary viscometer block temperature as outlined in Table 1 or Table 2. The programmed temperature is the temperature in Table 1 or Table 2 plus the appropriate temperature correction factor determined in **8.1**.

9.2 Test Sample and Viscometric Cell Preparation

9.2.1 Remove the nine rotors from the viscometric cells and ensure that both the cells and rotors are clean (*see* **9.6**) for the cleaning procedure.

9.2.2 Place a 10 ± 1.0 ml oil sample in each cell.

9.2.3 Install the rotors in the proper stators and install the upper pivots.

9.2.4 Place the loop of the 700 mm long string over the crossarm at top of the rotor shaft and wind all but 200 mm of the length of the string around the shaft. Do not overlap strings. Loop the remaining end of the string over the top bearing cover. Orient the rotor such that a (red or black) marked end of the crossarm at the top of the rotor shaft is pointing directly forward.

9.2.4.1 The string may be prewound around the shaft before installation of the rotor in step **9.2.3**.

9.2.5 Place the housing cover over the viscometric cells to minimize the formation of frost on the cold metal parts exposed to air. In some climates it may be necessary to flush the cover with dry air to minimize the frost formation.

9.2.6 Start the programmed temperature profile. Warm the oil samples to 80 ± 1°C and maintain at 80 ± 1°C for 2 h to allow solution of any material not in true solution at room temperature.

9.2.7 At the end of the 2 h soak at 80°C, the cooling cycle starts to cool the samples in accordance with the programmed cooling sequence as programmed in **9.1**.

9.2.8 At the completion of the temperature profile the temperature of the block should be within 0.2°C of the desired test temperature, when measured by a thermometer other than the temperature controller in the same thermometer well used during calibration. If the block temperature is within this range proceed with the yield stress and viscosity measurement within 30 minutes of the completion of the temperature profile (*see* **9.3**).

9.2.8.1 If the final temperature of the block is 0.2°C to 0.5°C warmer than the desired temperature, proceed as follows. Set the temperature controller to bring the block temperature to the correct test temperature and then hold at the correct test temperature for 30 minutes before proceeding. This entire temperature correction should not take longer than 1 h. The data obtained in this way are considered valid test results otherwise the test is invalid.

9.2.8.2 If the final test temperature is more than 0.2°C cooler or more than 0.5°C warmer than the preselected test temperature, then the test is not valid for the preselected temperature. For information only, the yield stress and viscosity may be measured without further temperature adjustment. These results are characteristic of the actual temperature, not the preselected one.

9.2.9 If the final temperature as noted in **9.2.8** is in error in either direction by more than 0.2°C:

a) Check the thermometer calibration.

b) Check the temperature sensor of the temperature controller for accuracy, in accordance with **8.1**.

c) Is the coolant flowing?

d) Replace methanol if wet, as indicated by ice-crytal in the top of the cold source reservoir, cold methanol absorbs water and as it absorbs water its cooling capacity decreases.

e) Is the bath refrigeration working properly?

f) Examine the temperature profile program for an error and make the appropriate corrections.

9.2.10 Inspect the recorded cooling profile data for temperature deviation greater than those temperature in Tables 1 and 2. Verify that the cooling rates permitted during the test are in conformance with those in Tables 1 and 2.

9.3 Measurement of the Yield Stress

9.3.1 Beginning with the cell farthest to the left of the instrument, follow the procedure below for each cell in turn.

9.3.2 Align the pulley wheel with the shaft of the cell to be tested, such that the string hangs past the front of the housing. Make sure that the weights clear the edge of the bench during testing.

9.3.3 Remove the string from the upper bearing support and carefully place it over the pulley wheel so as not to disturb the test cell. (Do not allow the rotor shaft to turn.)

9.3.4 Carefully, so as not to disturb the gel structure, attach a 10 g mass to the string and gently suspend the weight on the string. If the end of the crossarm does not move at least 3 mm in 15 seconds (approximately twice the diameter of the crossarm or 13° of rotation) then record that the sample has yield stress.

NOTE — When the 10 g load is first applied, some oils may show momentary movement of the crossarm. If there is no further movement of the crossarm for 15 s disregard the initial movement.

9.3.5 If yield stress is observed, proceed to **9.5**.

9.4 Measurement of Apparent Viscosity

9.4.1 Attach a 150 g mass to the string and slowly suspend the weight on the string. Start the timer when the crossarm of the rotor shaft points directly forward. If the first half-revolution requires less than approximately 10 second, measure and record the time required for three revolutions. If the time is greater than 10 second record the time for one revolution and identify it as the time for one revolution. If one revolution has not been completed in 60 seconds, end the measurement. Record as greater than 60 seconds for one revolution and do not calculate a viscosity. If the time for three revolutions is less than 4 seconds then record as less than 4 seconds.

9.5 Repeat **9.3** through **9.4.1** for each of the remaining cells in order from left to right.

9.6 Cleaning

9.6.1 After all of the cells have been completed turn off the cooling and turning on the heater to warm the viscometric cells to room temperature or somewhat higher. The temperature must not exceed 50°C.

9.6.2 Remove the upper rotor pivots and the rotors.

9.6.3 With a vacuum, remove the oil samples and rinse the cells with on oil solvent several times followed by two washings with acetone. Use a vacuum to remove the solvent from the cells after each rinse and allow the acetone to evaporate to dryness after the final rinse.

9.6.4 Clean the rotors in a similar manner.

10 APPARENT VISCOSITY CALCULATION

The viscosity is given by the following equation when using the cell constant obtained in **8.2.8**:

$$n_a = Ct\,3/r$$

where

n_a = apparent viscosity in cP (mPa.s),

C = cell constant obtained in **8.2.8**,

t = time for number (r) of complete revolutions of the rotor, and

r = number of revolutions timed.

11 REPORT

11.1 Apparent Viscosity and Yield Stress

Report the final test temperature and either the apparent viscosity or the existence of yield stress, but not both.

11.1.1 If the apparent viscosity is less than 5 000 mPa.s (cP) then report the apparent viscosity as less than 5 000 mPa.s (cP).

11.1.2 If the apparent viscosity is between 5 000 and 100 000 mPa.s (cP), then report the apparent viscosity to the nearest 100 mPa.s (cP).

11.1.3 If the apparent viscosity is between 100 000 and 400 000 mPa.s (cP), then report the apparent viscosity to the nearest 1 000 mPa.s (cP).

11.1.4 If the apparent viscosity is greater than 400 000 mPa.s (cP) then the apparent viscosity should be reported as greater than 400 000 mPa.s (cP).

12 PRECISION AND BIAS[1]

12.1 Precision

The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows.

12.1.1 *Yield Stress*

In the case of pass-fail data no generally accepted method for determining precision is currently available.

12.1.2 *Apparent Viscosity*

12.1.2.1 *Repeatability*

The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run in the normal and correct operation of the test method exceed the following values only in one case in twenty. The repeatability as a percent of the mean apparent viscosity is shown as follows:

Test Temperature °C	*Repeatability, Percent of Mean*
–15	4.2
–20	7.3
–25	11.7
–30	9.3
–35	20.7

12.1.2.2 *Reproducibility*

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only one case in twenty. The reproducibility as a percent of the mean apparent viscosity is shown as follows:

Test Temperature °C	*Reproducibility, Percent of Mean*
–15	8.4
–20	12.1
–25	17.5
–30	18.4
–35	21.5

12.1.3 The interlaboratory programme included nine test oils at the –15°C test temperature with eleven laboratories participating. Nine test oils were included at the –20°C test temperature with eleven laboratories participating. The –25°C test temperature included 18 test oils with 14 laboratories participating. Nine test oils were evaluated at –30°C in 13 laboratories. At the –35°C test temperature, nine sets of data were provided by seven laboratories on six test oils.

12.2 Bias

Since there is no accepted reference material suitable for determining the bias for this test method, no Statement on bias is being made.

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[1] Supporting data are available from ASTM headquarters. Request RR: D02 - 1212, D02 - 1249 and D02- 1277.

(Nonmandatory Information)

TEMPERATURE PROFILE FOR TEST TEMPERATURE

Table 1 Temperature Profile for Test Temperature –20°C and –35°C

(*Clauses* 4.2, 5.2, 5.2.1, 5.2.2, 8.4, 9.1 *and* 9.2.10)

Elapsed Time, A h:min	**Segment Time** h:min	**Segment Temperature, B**			**Allowable Temperature Variation C °C**
		Beginning °C	Final °C	Rate of Change °C/h	
(1)	(2)	(3)	(4)	(5)	(6)
0:20	0:20	above 20 to	80	—	—
2:20	2:00	80 to	80	—	± 1.0
2:40	0:20	80 to	0	—	—
2:43	0:03	0 to	–3.0	—	—
2:50	0:07	–3.0 to	–4.0	8.5	± 0.5
3:00	0:10	–4.0 to	–5.0	6.0	± 0.2
9:00	6:00	–5.0 to	–8.0	0.5	± 0.2
45:00	36:00	–8.0 to	–20.0	0.33	± 0.2
Hold at this point for –20°C test temperature D					
47:00	2:00	–20.0 to	–25.0	2.5	± 0.2
Hold at this point for –25°C test temperature D					
49:00	2:00	–25.0 to	–30.0	2.5	± 0.2
Hold at this point for –30°C test temperature D					
51:00	2:00	–30.0 to	–35.0	2.5	± 0.2
Hold at this point for –35°C test temperature D					

A To end of time segment.

B If the dual control loop concept is used, the bath set point temperatures should be 5°C below the corresponding block temperature desired. The maximum bath temperature shall not exceed –5°C.

C Holding the temperature variation to less than ± 1°C improves the precision and reproducibility of your viscosity measurements.

D The measurement of yield stress and apparent viscosity are to be made within 30 min of reaching the test temperature.

Table 2 Temperature Profile for Test Temperature –10° C and –15°C

(*Clauses* 4.2, 5.2, 5.2.1, 5.2.2, 8.4, 9.1 *and* 9.2.10)

Elapsed Time, A h:min	**Segment Time** h:min	**Segment Temperature, B**			**Allowable Temperature Variation C °C**
		Beginning °C	Final °C	Rate of Change °C/h	
(1)	**(2)**	**(3)**	**(4)**	**(5)**	**(6)**
0:20	0:20 above	20 to	80	—	—
2:20	2:00	80 to	80	—	± 1.0
2:40	0:20	80 to	10	—	—
2:43	0:03	10 to	7.0	—	—
2:50	0:07	7.0 to	6.0	8.5	± 0.5
3:00	0:10	6.0 to	5.0	6.0	± 0.2
9:00	6:00	5.0 to	2.0	0.5	± 0.2
45:00	36:00	2.0 to	–10.0	0.33	± 0.2
Hold at this point for –10°C test temperature D					
47:00	2:00	–10.0 to	–15.0	2.5	± 0.2
Hold at this point for –15°C test temperature D.					

A To end of time segment.

B If the dual control loop concept is used, the bath set point temperatures should be 5°C below the corresponding block temperature desired. The maximum bath temperature should not exceed –5°C.

C Holding the temperature variation to less than ± 1°C improves the precision and reproducibility of your viscosity measurements.

D The measurement of yield stress and apparent viscosity are to be made within 30 min of reaching the test temperature.

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